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(54) Title: FLUIDISED BED REACTOR

(57) Abstract: Gas-phase fluidised-bed reactor, suitable for polymerising one or more monomers, comprising a reaction chamber in the form of a vertical cylinder which at the bottom connects, via an open boundary plane, to an inlet zone the bottom of which is provided with an inlet and in which a fluidised bed can be maintained under operating conditions in a reaction zone, in which the reaction zone over a part of its height is divided into two or more compartments by one or more substantially vertical partition walls extending from a point located about the boundary plane.

WO 02/40146 A1

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For two-letter codes and other abbreviations, refer to the "Guid-ance Notes on Codes and Abbreviations" appearing at the begin-ning of each regular issue of the PCT Gazette.

FUIDISED BED REACTOR

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The invention relates to a fluidised-bed reactor for the polymerisation of olefinic monomers, comprising a reaction chamber, in the form of a vertical cylinder which at the bottom connects, via an open boundary plane, to an inlet zone the bottom of which is provided with an inlet and in which a fluidised

10   bed can be maintained under operating conditions in a reaction zone and to a process for the polymerisation of one or more monomers in a fluidised-bed reactor, which reactor comprises a reaction chamber in the form of a vertical cylinder which at the bottom connects, via an open boundary plane, to an inlet zone the bottom of which is provided with an inlet, comprising the maintenance of

15   a fluidised bed in a reaction zone within said reaction chamber, withdrawing at least part of the gaseous stream from the top of the reactor and recycling that stream to the reactor via the inlet.

Gas-phase fluidised-bed polymerisation of one or more monomers, like an olefin or olefins, is known per se, as are suitable reactors for

20   that polymerisation. The polymerisation is effected in a usually vertical elongated reactor in which a bed of polymer particles is maintained in fluidised condition with the aid of an ascending gas stream which contains at least the gaseous monomer(s) to be polymerised. It is noted that the fluidised bed extends only over part of the height of the reaction chamber. The area in which the fluidised bed is

25   present is designated as the reaction zone. Most known reactors are fed by means of a gas distribution plate that separates the bottom section of the reactor from the reaction zone proper. This plate has been provided with openings that distribute the supplied gas stream across the area of the reaction zone as required.

30   The ascending gas stream may optionally contain one or more inert gases and for example hydrogen as a chain length regulator. An important objective of the addition of inert gases is to control the dew point of the gas mixture. Suitable inert gases are for example inert hydrocarbons such as (iso)butane, (iso)pentane and (iso)hexane, but also nitrogen. Such an inert gas

35   may be added to the gas stream as a gas or, in condensed form, as a liquid.

The gas stream is discharged via the top of the reactor and, after certain processing operations, fresh monomer is added to it to make up for

the monomer(s) consumed in the polymerisation, and then the gas stream is again supplied to the reactor as (a portion of) the ascending gas stream in order to maintain the bed.

5   A catalyst is also added to the bed. During the process, under the influence of the catalyst present, fresh polymer is continuously formed and at the same time polymer that has formed is withdrawn from the bed, with the bed volume and mass being kept substantially constant.
Converted monomer is continuously made up by means of the gas stream that maintains the bed.

10 From DE-19821955-A1 a reactor as described in the preamble is known. The feature that distinguishes this reactor from the reactors known until then is the total absence of a gas-distribution plate that separates the inlet zone from the reaction chamber or the presence at the location of the inlet or at the bottom of the inlet zone of a gas- distribution plate with a large open surface area,

15 in any case more than 20% of the total surface area. This document further teaches that it is preferred to provide the reactor with means that ensure that the supply to the fluidised bed of the reaction components fed through the inlet is as homogeneous as possible. This same purpose was achieved in the reactors known until then by the presence of a gas-distribution plate featuring a large

20 number of small openings uniformly distributed over the gas-distribution plate. Thus, the reaction components supplied are homogeneously distributed over the boundary plane while being fed to the reaction chamber.

A disadvantage of the known reactor is that at a certain diameter of the fluidised bed the height of the fluidised bed can be no more than 3 to 5

25 times said diameter. This limits the capacity of the reactor.

The object of the invention is to provide a reactor in which a stable fluidised bed can be maintained with a higher height – diameter ratio than in the known reactor.

This object is achieved in that the reaction zone over a part of its

30 height is divided into two or more compartments by one or more substantially vertical partition walls extending from a point located about the boundary plane.

The reactor according to the invention having at least one partition wall has the additional advantage that it is now possible to choose a higher H/D ratio for fluidised bed in the reactor, for instance an H/D ratio of greater

35 than 5, and even up to 20, which is much higher than in the case of the known reactors, while yet maintaining a stable fluidised bed, resulting in a more

controlled polymerisation process. This advantage makes it possible to apply slimmer reactor vessels which implies major engineering advantages for polymerisation reactors because they are pressure vessels.

5   A further advantage of the reactor according to the invention is that the gas stream discharged from the top of the reactor contains fewer fine polymer particles.

It has also been found that in the reactor according to the invention bigger particles can be kept in fluidised condition than in the known reactor without partition wall.

10   The polymerisation is an exothermic reaction. Heat needs to be removed continuously so as to keep the temperature in the reactor at the desired level. Such removal is effected via the gas stream, which leaves the reactor at a higher temperature than that at which it is supplied to the reactor. The superficial gas velocity in the reactor cannot be chosen to be arbitrarily large and so no

15   arbitrarily large amount of heat can be removed. The minimum velocity is dictated by the requirement for the bed to remain fluidised. On the other hand, the velocity must not be so large that a significant amount of polymer particles is blown out through the top of the reactor. The aforementioned limits are strongly dependent on the dimensions and the density of the polymer particles present in the bed and

20   can be determined by experiment. Practical values for the superficial gas velocity are between 0.05 and 1.0 m/sec. These requirements are elements that limit the maximum flow rate of the gas stream at the given reactor dimensions and, thus, the maximum attainable heat removal. The maximum allowable amount of heat of reaction produced, and hence the maximum amount of polymer to be produced,

25   are limited likewise.

The detailed design and operation of fluidised-bed reactors for the polymerisation of one or more olefin monomers and suitable process conditions are known per se and are described in detail in for example US-A-4,543,399 and in WO-A-94/28032.

30   From that same US-A-4,543,399 it is known to make up the gas stream discharged from the reactor with fresh monomer(s) and to cool it to a point where the stream partly condenses (the so-called "condensed mode"). The two-phase stream so obtained, which because of the latent heat of evaporation of the liquid phase has a substantially larger heat removal capacity, and so a

35   corresponding cooling capacity, than a stream consisting solely of a gas, is recycled to the bottom of the reactor. The dew point of the two-phase stream must

be lower than the temperature in the reaction zone so that the liquid can evaporate in it. In this way, the production capacity of a fluidised-bed reactor appears to be substantially higher than that of reactors which use a recycle gas without condensed liquid, said reactor having otherwise equal dimensions. In the

5   known process the maximum amount of liquid in the two-phase stream is 20 wt. %. The highest figure quoted in the examples is 11.5 wt. %.

From WO-A-94/28032 it is known to separate the liquid from the two-phase stream obtained on cooling of the gas stream to be recycled and to feed said liquid to the reactor separately from the gas stream. The liquid is

10   preferably injected or atomised at a certain height into the fluidised bed proper, optionally with the aid of a gaseous propellant. In this way, according to this publication, it is possible to feed a larger amount of liquid in proportion to the amount of gas being fed. This allows an even larger amount of heat to be removed, so allowing higher polymer production with proportionally higher heat

15   production. WO-A-94/28032 quotes a figure of 1.21 as the maximum permissible ratio of the mass of liquid feed to the mass of the total gas feed, which figure was derived from a simulated experiment.

It has been found that when in the reactor according to the invention a fluidised bed is maintained that extends, both at the top and at the

20   bottom, beyond the partition walls, so that the partition walls are submerged in the fluidised bed, more liquid can be supplied in proportion to the total gas feed than in the absence of a partition wall. This increases the heat removal capacity of the process, thus allowing higher heat production and hence higher polymer production rates at equal reactor dimensions. Even at a constant liquid to gas

25   mass ratio in the feed to the reactor, the process of the present invention results in a higher productivity of the reactor.

The bottom of the reaction chamber connects to an inlet zone via an open boundary plane. An open boundary plane is understood to be a boundary plane in which no gas-distribution plate is present and which is at least

30   90% and preferably fully permeable to the reaction components and the polymer powder in the fluidised bed.

The inlet zone is connected to the boundary plane and is provided with an inlet opening at the bottom. The inlet zone preferably has the form of a truncated cone so that the inlet zone becomes uniformly wider from the

35   inlet up to the boundary plane. The angle of the wall of the inlet zone to the boundary plane is preferably at least 30° and preferably at most 75°. The height of

the inlet zone, that is the perpendicular distance between the plane of the inlet and the boundary plane, is preferably between 0.3 and 2 times the diameter of the reaction chamber. The surface area of the inlet opening is between 2.5 and 25% of the surface area of the boundary plane and preferably between 5 and 20 %

5   thereof.

In particular, the reactor of the present invention advantageously forms part of a reactor system, which further comprises means for recirculating the stream out of the cooler/condenser to the reactor as a gas/liquid mixture. In another preferred mode, the reactor system also comprises a gas-liquid separator

10 to separate at least part of the condensed liquid out of the resulting two-phase stream from the cooler/condenser and means for introducing at least part of the separated liquid into the fluidised-bed reactor.

A particularly suitable partition wall in the reactor of the invention is a pipe or hollow section placed in a vertical position, preferably concentric with

15 the reactor. Since the pipe or hollow section is completely submerged in the fluidised bed, no appreciable pressure differences occur across the wall of the pipe so that the pipe may be of light-duty construction. This applies also to walls of different shapes.

The walls can simply be suspended from a higher section of the

20 reactor, supported by a bottom section or secured to the wall of the reactor. In the present context a hollow section differs from a pipe in terms of the shape of its cross-section. The cross-section of a pipe is curved, for example circular or elliptical, whilst that of a hollow section is angular, for example triangular, rectangular, octagonal or with more angles, with or without the angles being

25 uniformly divided. The hollow section or the pipe may have a uniform and/or tapered cross-section, for instance a cone shape, including tapering inwardly and outwardly, for instance, in a hyperbolic shape. For conical shapes, it is preferred that the apex angle formed by the walls of pipe or hollow section is generally not more than 5°, preferably not more than 2.5°. Particularly suitable are angles

30 between 0° and 2°. The ratio of the area of the radial cross-section of the pipe or hollow section to that of the reactor is between 1:9 and 9:10 and, in order to achieve as high a stability as possible, preferably between 1:5 and 3:4. In the case of a conical pipe or hollow section, the same applies to the average cross-sectional area thereof. The vertical partition wall extends from a point located

35 about the boundary plane to a point located below the top of the reaction zone.

'About' in this respect means that the partition wall extends upward from the boundary plane or from a point situated at at most 0.5 and preferably at most 0.25 times the diameter of the reaction chamber above it. In case a pipe or hollow section is applied the smallest distance between the lower edge of the pipe or

5   hollow section and the nearest point of the side wall of the inlet zone is preferably at least equal to the distance between the pipe and the side wall of the reaction chamber.

If the dimensions given here are departed from, the favourable effect of the presence of a vertical partition wall is diminished. The upper end is located at

10   least 0.1 x the diameter of the reaction chamber below the end of the reaction zone and preferably not more than 3 x that diameter.

It has been found that it is far less critical for the bed to extend further beyond the partition wall at the upper end than at the lower end. The upper end of the partition wall may be lower accordingly as the H/D ratio of the fluidised bed increases.

15   What is stated here on the positioning of the wall in the reaction zone applies also to the vertical partition walls to be explained below.

          · Another embodiment of a suitable partition wall is a substantially axially oriented flat, curved or folded plate present in the reaction zone. It is preferred for such a partition wall to extend from one vertical line of the inner wall

20   of the reactor chamber to an other line although a clearance of up to 10 cm between the partition wall and side wall is permissible. In this way, the reaction zone is divided into two or more compartments, which may be differently sized. The area ratio of the radial cross-section of a compartment to the radial cross-section of the reactor preferably is between 0.1 and 0.9 and more preferably

25   between 0.20 and 0.75. The substantially axially oriented wall should be virtually vertical. This should be understood to mean parallel with the axis of the reactor in its vertical position but also out of parallel by not more than 5°, preferably not more than 2.5°.

              The aforementioned beneficial effects of a partition wall occur

30   when there is a common inlet for a gas/liquid mixture at the underside of the reactor, as described in US-A-4,543,399, and also when there is a separate gas and liquid inlet in the fluidised bed, as described in WO-A-94/28032.

              In the latter case, the liquid may be supplied to the fluidised bed via the underside of the reactor at one or more points through the wall of the inlet

35   zone as well as at one or more points through the side wall of the reaction

chamber. It is in any case advantageous to arrange the means of introduction of the liquid in such a way that the bulk of the liquid can be supplied into the fluidised bed in a zone located under or in the central compartment if a pipe or hollow section is employed, or under or into one of the compartments if one or more

5   vertical partition walls are present. In the case of introduction of the liquid via one or more points through the side wall of the reactor chamber or of the inlet zone, and if the partition wall is a pipe or hollow section, it is advantageous to position the means of introduction so that the liquid can be supplied to the fluidised bed at a point below the lower end of the pipe or hollow section. In that case, for example

10 by suitably choosing the feed velocity, the liquid can be supplied to both the central compartment and the peripheral compartment of the reactor. Preferably, the bulk of the liquid is supplied to the central compartment, located within the pipe or hollow section, inasmuch as the best results are obtained herewith.

          In the case that a vertical plate is used as a partition wall, the

15 liquid may be introduced in the aforementioned manner from a height below the lower end of the wall but also via inlets arranged at different heights in the section of the reactor wall which confines the compartment or compartments to which the liquid is to be supplied. In this case the liquid is preferably supplied to that compartment in which the ratio between 1) the part of the inlet area that is below

20 said compartment viewed from the direction of the reactor axis and 2) the surface area of said compartment, is highest. When this ratio is the same for the two compartments, the bulk of the liquid can be supplied to one of the two compartments as desired.

          In reactors with a height / diameter ratio of more than 5, means

25 for the supply of the liquid can also be positioned above the top of the partition wall, if the distance from this top to the top of the fluidised bed is at least about 2 m.

          The invention further relates to a process for the polymerisation of one or more monomers in a fluidised-bed reactor, which reactor comprises a

30 reaction chamber in the form of a vertical cylinder which at the bottom connects, via an open boundary plane, to an inlet zone the bottom of which is provided with an inlet, the process comprising the maintenance of a fluidised bed in a reaction zone within said reaction chamber, withdrawing a gaseous stream from the top of the reactor and recycling at least part of that stream to the reactor via the inlet,

35 wherein part of the reaction zone is divided into two or more compartments by one or more substantially vertical partition walls extending from a point located about

the boundary plane to a point located below the top of the reaction zone and wherein the fluidised bed extends above and below the partition walls.

Conducting the process in the reactor with the specified characteristics brings about all the advantages associated with such a reactor as described herein before, both when the process is a condensed mode process and when it is a classical gas phase process. In the process of the invention the reactor can be operated in a stable manner even when the mass ratio of (liquid supplied to the reactor): (amount of gas supplied to the reactor) is higher than 2:1 or even higher than 4:1. The aforementioned ratio is in any case at least 10% and even more than 50% to even more than 100% higher than when the process is operated in a similar reactor without partition wall(s).

The liquid is preferably injected in finely divided form, preferably in atomised form, optionally with the aid of a propellant, for which purpose for example recycle gas or fresh monomer gas may be used. Injection should take place in such a way that the liquid enters the desired compartment whence it is taken up by an ascending fluidising gas stream. This has been found to be favourable in terms of the amount of liquid that can be supplied to the fluidised bed without sintering of polymer particles or other undesired disturbances occurring in the bed.

In case a condensed mode process is applied, i.e. that part of the recycle is introduced in liquid form into the reactor, introducing the liquid via several inlets at different heights of the reactor gives the possibility to vary the concentration of the different ingredients of the liquid inlet (through the addition of more or less make-up monomer, etc.) which improves the operating window of the polymerisation reaction and therefore broadens the product capabilities of the fluidised bed reactor.

The supplied amount of gas includes, besides the gas supplied via the recycle stream, all other gases supplied to the reactor, including at least the propellant and carrier gases that are employed in introducing the catalyst, a catalyst activator and/or other substances desired or needed for the polymerisation and those used for atomising the supplied liquid.

The process according to the present invention is suitable for any kind of exothermic polymerisation reaction in the gas phase. Suitable monomers include olefin monomers, polar vinyl monomers, diene monomers and acetylene monomers. The process of the present invention is especially suitable for the manufacture of polyolefins by the polymerisation of one or more olefin

monomers, at least one of which is preferably ethylene or propylene. Preferred olefin monomers for use in the process of the present invention are those having from 2 to 8 carbon atoms. However, small quantities of olefin monomers having more than 8 carbon atoms, for example 9 to 18 carbon atoms, can be employed if

5   desired. Thus, in a preferred mode, it is possible to produce homopolymers of ethylene and/or propylene or copolymers of ethylene or propylene with one or more C_2-C_8 alpha-olefin monomers. The preferred alpha-olefin monomers are ethylene, propylene, butene-1, pentene-1, hexene-1, 4-methylpentene-1, and octene-1. An example of a higher olefin monomer that

10 can be copolymerised with the primary ethylene and/or propylene monomer, or as partial replacement for the C_2-C_8 monomer, is decene-1. Dienes are also suitable, like 1,4-butadiene, 1,6-hexadiene, dicyclopentadiene, ethylidene norbornene and vinyl norbornene.

     When the process is used for the copolymerisation of ethylene

15 and/or propylene with other alpha-olefin monomers, the ethylene and/or propylene are present as the major component of the copolymer, and preferably are present in an amount at least 70 wt %, more preferably 80 wt. % of the total monomers.

     The process is particularly suitable for polymerising olefin monomers at a pressure of between 0.5 and 10 MPa, preferably between

20 1 and 5 MPa, and at a temperature of between 30°C and 130°C, and particularly between 45°C and 110°C.

     The polymerisation reaction may be carried out in the presence of any catalyst system known in the art (for instance anionic catalyst, cationic catalyst or free-radical catalyst) suited for the gas-phase polymerisation of one or

25 more (olefin) monomers, like a catalyst system of the Ziegler-Natta type, consisting of a solid catalyst essentially comprising a compound of a transition metal and of a co-catalyst comprising an organic compound of a metal (i.e. an organometallic compound, for example an alkyl aluminium compound); so-called single site catalyst systems, like metallocene-based catalyst systems, are also

30 suitable.

     The catalyst may also be in the form of a prepolymer powder prepared in a prepolymerisation stage with the aid of a catalyst system described above. The prepolymerisation may be carried out by any known process, for example polymerisation in a liquid hydrocarbon diluent or in the gas phase using a

35 batch process, a semi-continuous process or a continuous process.

It should also be appreciated that the present invention is suitable for retrofitting existing reactors by installing one or more partition walls, pipes or hollow sections in the reactor. In particular, a reactor could be retrofitted by installing a pipe, as discussed above, by fixedly attaching the pipe to an

5   internal section of the reactor.

The invention is applicable both to so-called "grass roots" installations as well as for de-bottlenecking existing fluidised bed polymerisation installations. In the last case the full benefit of the invention might not be obtainable, as the capabilities of other units in the total polymerisation system may

10   form a constraint on the maximum productivity of the system. (In other words: the throughput of the polymerisation system as a whole might be hindered by constraints in the system other than in the reactor section.) In situations where a new, integrated, polymerisation plant is designed and built ("grass roots"), the benefits of the present invention can be fully used and exploited.

15   The invention is elucidated by means of the following drawings, which are not intended to limit the invention.

In these drawings Fig. 1 is a set-up for polymerising one or more monomers, which includes a first embodiment of the process of the present invention with a reactor having a tubular partition wall and with a common inlet for gas and liquid;

20   Fig. 2 is a similar set-up which includes a second embodiment of the process of the invention in which gas and liquid are separately supplied to the reactor;

Fig. 3 is a set-up as in Fig. 2, in which liquid is supplied through the wall of the reaction compartment and in which the tubular partition wall has a conical shape;

Fig. 4 is a set-up as in Fig. 3 with a vertical plate serving as partition wall and in

25   which liquid is introduced through the reactor wall at different heights;

Fig. 5 is a radial cross-section of the reactor in Fig. 4 along line A-A; and

Fig. 6 is a similar cross-section of a reactor in which a folded vertical plate serves as partition wall.

In the various drawings numbers that correspond in their 2 last figures denote

30   corresponding items.

Fig. 1 shows a reactor chamber 2 in the form of a vertical cylinder in which 4 is an inlet zone which connects to the reaction chamber proper 2 at the location of the boundary plane.

Fluidising gas is introduced into the inlet zone 4 through inlet opening 10 via inlet

35   line 8. The surface area of inlet opening 10 may be between 2.5 and 25% of the surface area of the boundary plane and is preferably between 5 and 20% thereof.

One skilled in the art will know how to choose the size of the inlet opening in
relation to the flow rate of the gas feed and the limiting values for the required gas
velocities needed for maintaining a stable fluidised bed.

In the reaction zone 12 and the upper part of the inlet zone 4 the gas stream
5   introduced maintains a fluidised bed of growing polymer particles which extends to
below or even to the bottommost part of velocity-reducing zone 14. A cylindrical
pipe 16 is concentrically suspended in reaction zone 12 from the wall of reactor
chamber 2 with supports 18. Pipe 16 is submerged in the fluidised bed. Zone 14
widens relative to reaction zone 12. In this zone 14 the gas velocity decreases to
10   the point where the gas is unable to substantially entrain any further the polymer
particles that have formed in the reaction zone. As a consequence, the recycle
stream discharged through discharge line 20 is virtually free from entrained
polymer particles. The recycle stream is cooled in heat exchanger 22,
compressed in compressor 24 and cooled in heat exchanger 26 to a temperature
15   such that a proportion of the recycle stream condenses to form a two-phase
stream. Make-up monomer is added to this two-phase stream through line 28,
whereupon the gas-liquid mixture is reintroduced via opening 10 into the inlet
zone 4 of the reactor through opening 10. A polymer-gas stream is discharged
from the reactor through drain line 30, which can be closed by valve 32. This
20   stream is separated into polymer and gaseous components in separator 34. The
polymer is discharged from separator 34 via the bottom and is processed further.
Such further processing may involve processes that are known per se and are not
shown in the figure, like removal of absorbed or dissolved liquid.

The gaseous components, on being pressurised to the required
25   pressure, are added to the recycle stream in line 20 via line 35. Besides the
monomer, the required catalyst system and, optionally, an activator are supplied
to the reactor. Here, too, the catalyst system is preferably supplied directly to the
fluidised bed above the boundary plane from storage vessel 36 and propelled by
an inert gas through line 38, which also terminates below the lower end of pipe 16.
30   The activator can be added to the feed stream in line 8 via metering device 40. If
necessary, a propellant is also added to this stream via feed line 42.

The set-up in Fig. 2 differs from the one in Fig. 1 in that the two-
phase stream that is formed in the second cooling step in heat exchanger 226 is
passed to gas-liquid separator 244. In this separator, gas and liquid in the two-
35   phase stream are separated from each other. After addition of make-up monomer
and, optionally, inert gas via the feed lines 228 and 242, respectively, the

separated gas stream is supplied to the inlet zone 204 via inlet line 208. The
separated condensed liquid stream is supplied to the fluidised bed via atomiser
246 connected to line 248 that extends through the side wall of inlet zone 204.

5   In Fig. 3, contrary to Fig. 2, the liquid separated in gas-liquid
separator 344 is supplied to the fluidised bed via a number of lines whose ends
are symmetrically arranged along the circumference of the reactor wall at a height
between the boundary plane 306 and the lower end of pipe 316. Two such lines,
350 and 352, are shown in the figure. The lines pass through the reactor wall into
reaction zone 312 and are terminated with atomisers 354 and 356. Through these

10   atomisers the liquid, finely atomised with the aid of fresh monomer as propellant,
is introduced into the fluidised bed at such an exit velocity that the liquid is
entrained by the fluidised bed through the central compartment located within pipe
316. Pipe 316 also has a conical shape here with an apex angle of 1.5°. For the
sake of clarity, the conical shape shown is exaggerated.

15   In Fig. 4, 460 is a vertical plate which serves as a partition wall
to divide the reactor into two unequal compartments 462 and 464. A cross section
of this reactor is shown in Fig. 5. In Fig. 4 a number of liquid inlets 466 pass
through the reactor wall at different heights to terminate in compartment 462.
Catalyst feed line 438 also terminates in this compartment. In Fig. 6, the partition

20   wall plate 660 is folded.

The invention is further elucidated by means of the following
computer-simulated Examples and comparative experiments, which are not meant
to restrict the invention thereto.

25   Examples I-IV and comparative experiments A and B

A continuous polymerisation of propylene to polypropylene is
performed in a vertical, cylindrical fluidised-bed reactor with an inner diameter of
0.85 m. The distance between the boundary face and the top of the reactor is 8.5
m; the fluidised bed has a height of 4.2 m. The angle between the wall of the inlet

30   zone and the boundary plane is 45°. The diameter of the inlet opening is 0.17 m.

As catalyst system, a fourth-generation heterogeneous
Ziegler/Natta catalyst system is used, the catalyst having an average particle size
of 20 μm.

In all the Examples, a concentric cylindrical pipe with a diameter

35   of 0.45 m, a length of 3.2 m, and a wall thickness of 2×10^{-3} m, is placed in the

fluidised bed, at 0.2 m above the boundary plane. This pipe is absent in the comparative experiments.

The catalyst system, propylene, hydrogen, nitrogen (and an inert coolant) are continuously fed to the reactor; the off-gas of the reactor is cooled to

5   a temperature below its dew point and recirculated to the bottom of the reactor. A stream of polymer product is withdrawn from the bottom of the fluidised bed. The superficial gas velocity in the fluidised bed is maintained at 0.6 m/s.

During the polymerisation the maximum percentage of condensed mode (MCM, in %) is determined; this is the point at which operation

10   of the reaction, and especially the reaction temperature, becomes unstable, and polymer withdrawal problems are encountered. The condensed mode ratio is the ratio of the weight of the liquid versus the weight of gas and liquid in the two-phase recycle stream to the reactor. The percentage of condensed mode can be varied by changing the cooling temperature of the recycle stream.

15   The process conditions and the resulting MCM and reactor productivity are given in Table I.
In Examples I and III and comparative experiments A and B propylene is used as the condensable agent; in Example II a mixture of propylene and isobutane (IB) is used as the condensable agent; in Example IV a mixture of propylene and

20   isopentane (IP) is used as the condensable agent. The composition of the gas phase is monitored in-line by means of gas chromatography.

Table I

Example/ comp. exp.	Total pressure (MPa)	$C_3^=$ pressure (MPa)	H_2 pressure (MPa)	N_2 pressure (MPa)	IB/IP Pressure (MPa)	Reactor temp. (°C)	MCM (%)	Recycle temp. (°C)	Productivity (t/h)
I	2.50	2.16	0.04	0.30	-	72	65	36	5.0
II	2.50	2.16	0.04	0.20	0.10	72	61	47	4.8
III	2.30	1.99	0.04	0.28	-	72	63	34	4.8
IV	2.30	1.99	0.04	0.07	0.21	72	59	43	4.7
A	2.50	2.16	0.04	0.30	-	72	29	45	2.0
B	2.30	1.99	0.04	0.28	-	72	28	42	1.9

From the data it can be seen that use of a process of the present invention, and more specifically use of the reactor system of the present invention, results in a sharp increase in the applicable condensed mode, as a result of which a much higher reactor productivity is achieved.

5

CLAIMS

1.   Gas-phase fluidised-bed reactor, suitable for polymerising one or more
     monomers, comprising a reaction chamber in the form of a vertical
     cylinder which at the bottom connects, via an open boundary plane, to an
     inlet zone the bottom of which is provided with an inlet and in which a
     fluidised bed can be maintained under operating conditions in a reaction
     zone, characterised in that the reaction zone over a part of its height is
     divided into two or more compartments by one or more substantially
     vertical partition walls extending from a point located about the boundary
     plane.

2.   Reactor according to Claim 1, characterised in that the partition wall is a
     pipe or hollow section.

3.   Reactor according to Claim 2, characterised in that the pipe or hollow
     section is concentric with the reactor.

4.   Reactor according to any one of Claims 1-3, characterised in that the H/D
     ratio of the reactor is greater than 5.

5.   Reactor according to any one of Claims 1-4, characterised in that the
     ratio of the area of the radial cross-section of the pipe or hollow section to
     that of the reactor is between 1:5 and 3:4.

6.   Reactor according to Claim 1, characterised in that the partition wall is a
     substantially axially oriented flat, curved or folded plate.

7.   Reactor system comprising a reactor according to any one of Claims 1-6,
     characterised in that the reactor system further comprises means for
     recirculating the stream out of the cooler/condenser to the reactor as a
     gas-liquid mixture.

8.   Reactor system comprising a reactor according to any one of Claims 1-6,
     characterised in that the reactor system further comprises a gas-liquid
     separator to separate at least part of the condensed liquid out of the
     resulting two-phase stream from the cooler/condenser and means for
     introducing at least part of the separated liquid into the fluidised bed
     reactor.

9.   Process for the polymerisation of one or more monomers in a fluidised
     bed reactor, which reactor comprises a reaction chamber in the form of a
     vertical cylinder which at the bottom connects, via an open boundary
     plane, to an inlet zone the bottom of which is provided with an inlet,

comprising the maintenance of a fluidised bed in a reaction zone within said reaction chamber, withdrawing at least part of the gaseous stream from the top of the reactor and recycling that stream to the reactor via the inlet, characterised in that part of the reaction zone is divided into two or more compartments by one or more substantially vertical partition walls extending from a point located about the boundary plane to a point located below the end surface of the reaction zone.

10.   Process according to claim 9 in which at least part of the gaseous stream withdrawn from the top of the reactor is cooled to a point where the stream partially condenses into a liquid and in which at least part of the condensed liquid is recycled to the reactor.

11.   Process according to Claim 9, characterised in that the partition wall has the shape of a pipe or hollow section.

12.   Process according to Claim 11, characterised in that the pipe or hollow section is concentric with the reaction zone.

13.   Process according to Claim 9, characterised in that the partition wall is a substantially axially oriented flat, curved or folded plate.

14.   Process according to any one of Claims 10-13, characterised in that the resulting two-phase stream is recycled to the reactor as a gas-liquid mixture.

15.   Process according to Claim 10, characterised in that at least part of the condensed liquid is separated from the two-phase stream and directly introduced into the fluidised bed.

16.   Process according to any one of Claims 14-15, characterised in that the mass ratio of (liquid supplied to the reactor) : (the amount of gas supplied to the reactor) is higher than 2:1.

17.   Process according to any one of Claims 9-16, characterised in that the H/D ratio of the fluidised bed is greater than 5.0.
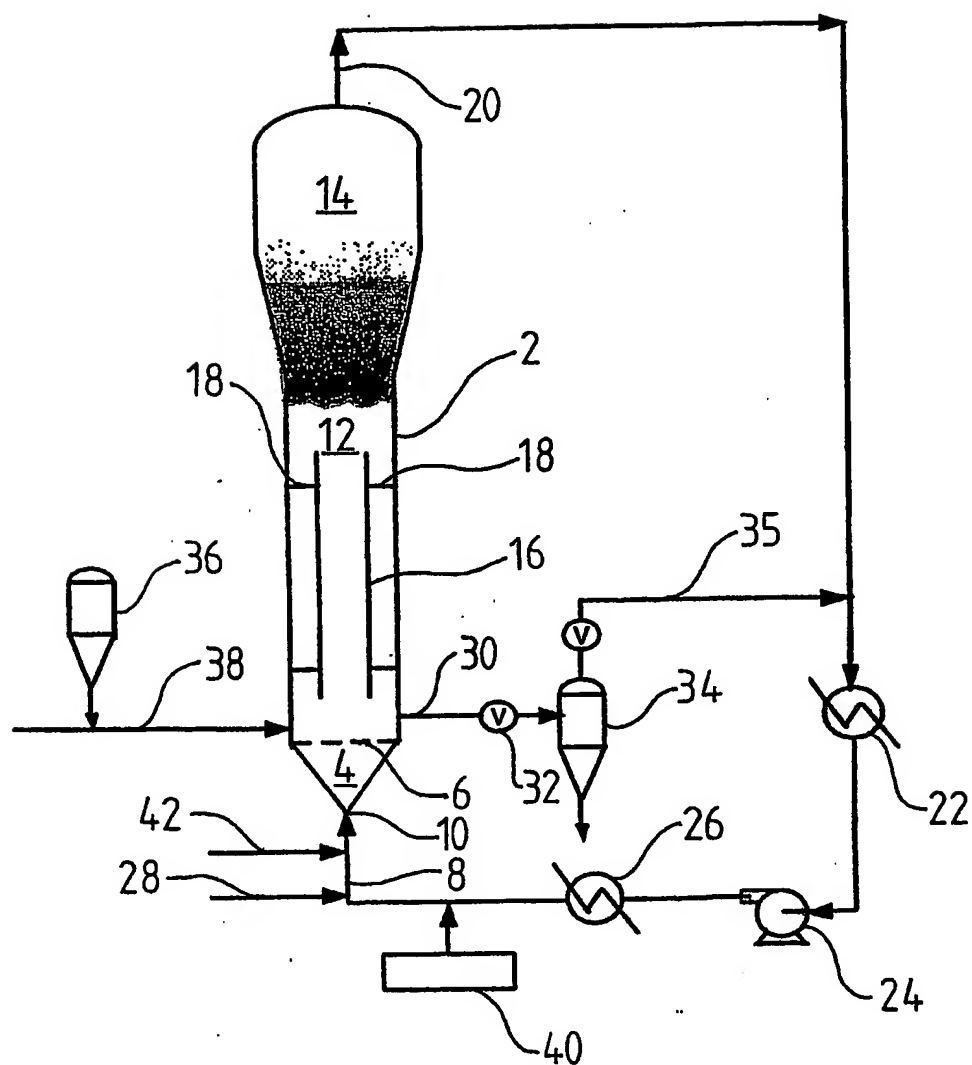
Fig. 1

Fig. 2
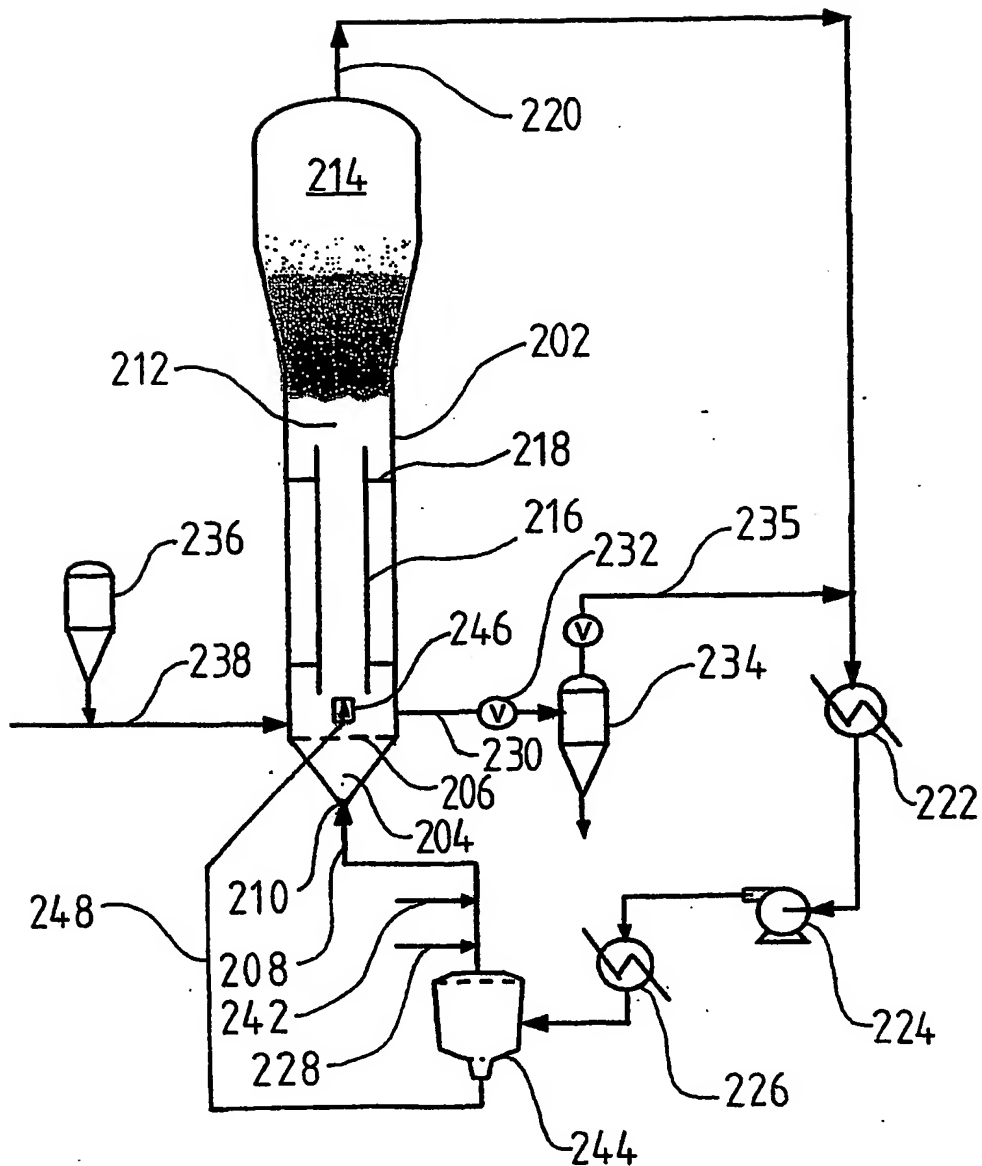
Fig. 3
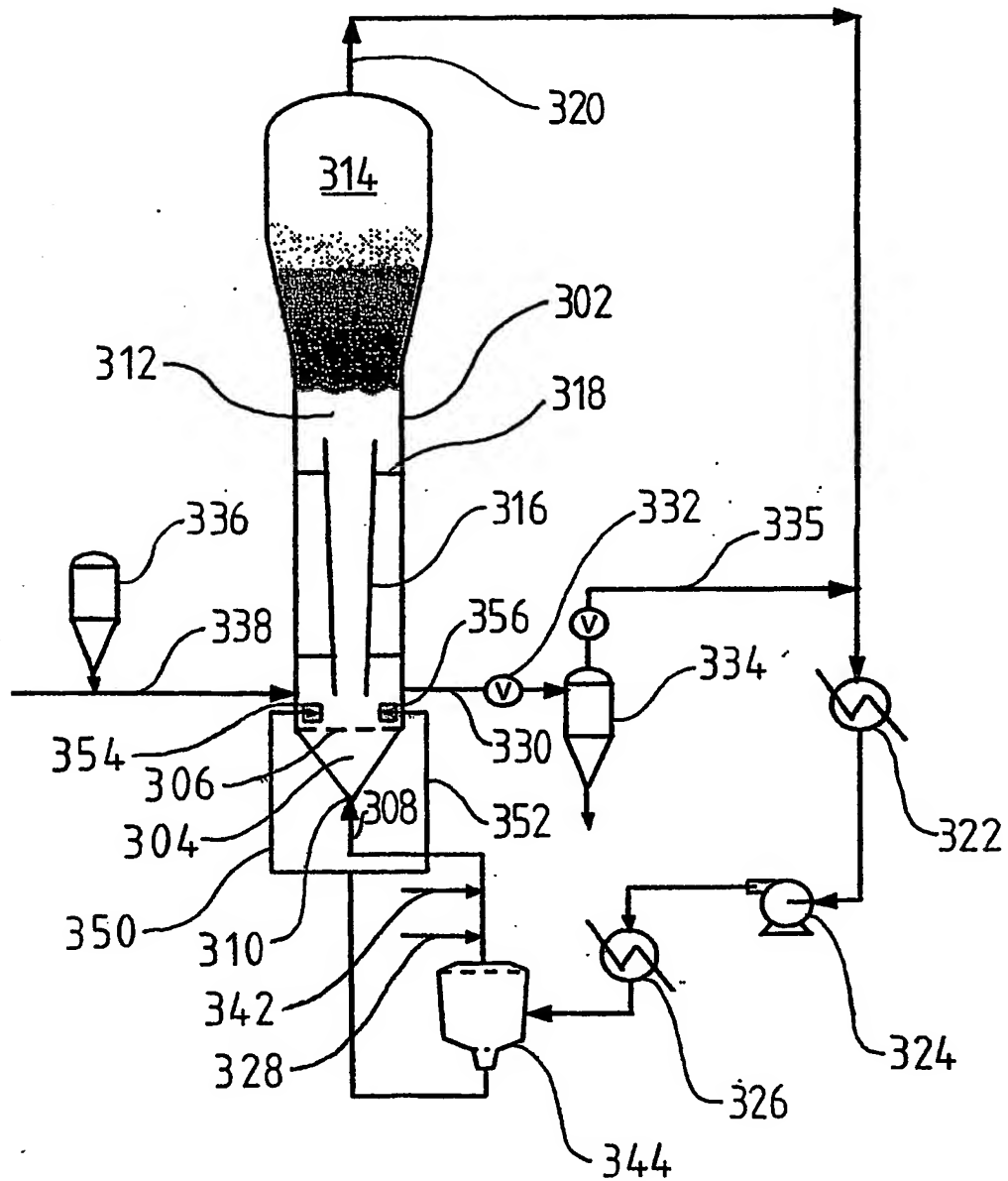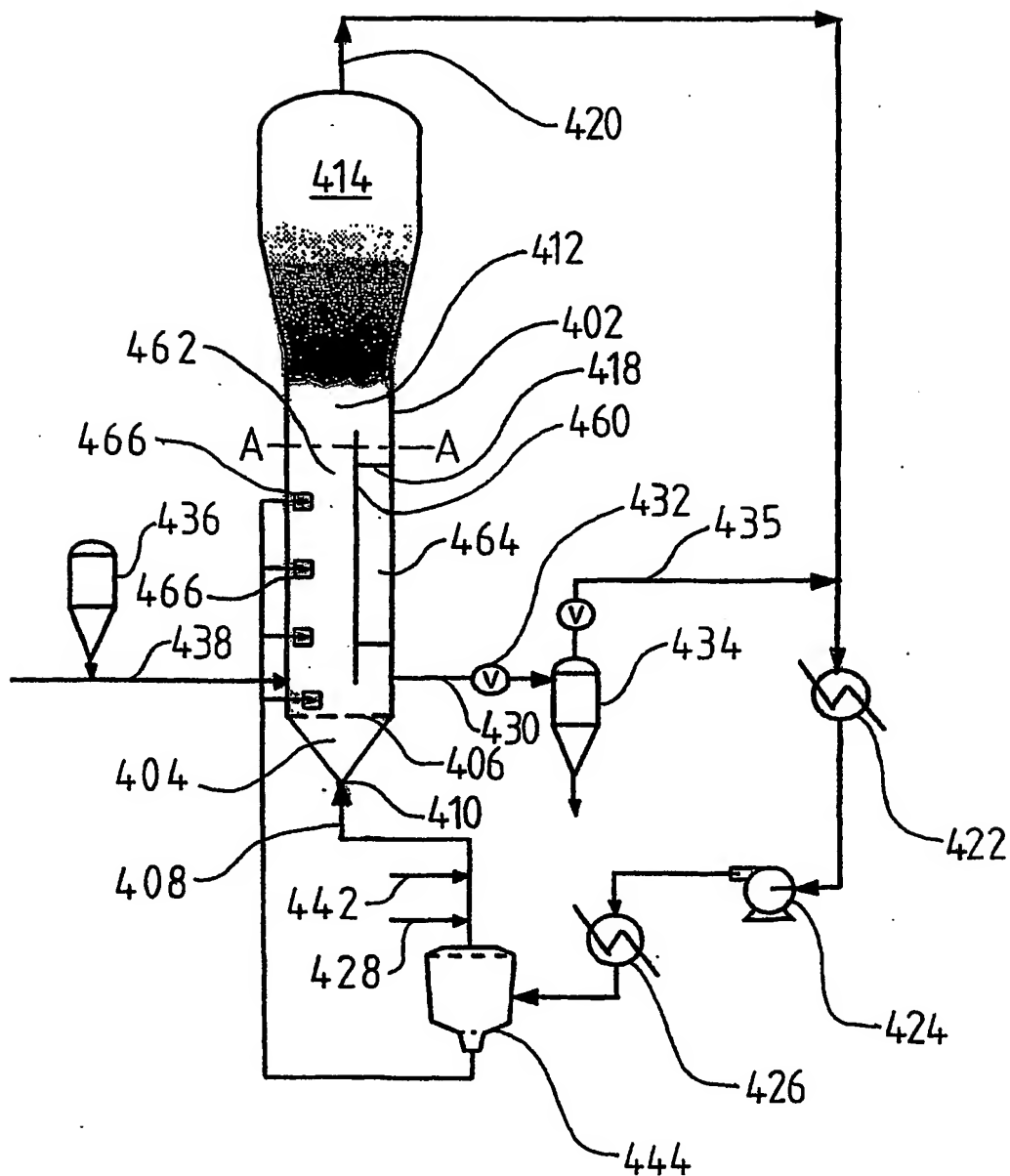
Fig. 4

5/5
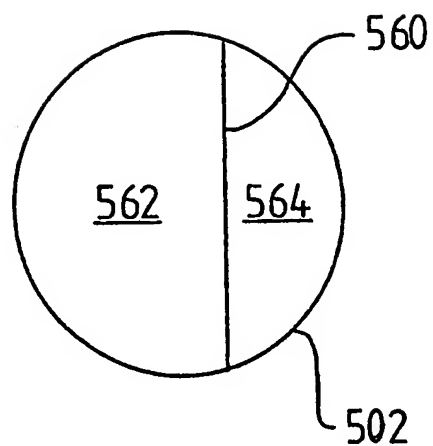
Fig. 5

Fig. 6

INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7   B01J8/34   B01J8/24   C08F10/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7   B01J C08F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E	WO 00 69552 A (DSM NV ;MUSTERS STANISLAUS MARTINUS PE (NL)) 23 November 2000 (2000-11-23) the whole document	1-17
X	US 5 114 700 A (MEIHACK WOLFGANG F A T ET AL) 19 May 1992 (1992-05-19) column 4, line 51 -column 6, line 26; figures 1-6	1-3,6, 9-13
X	US 4 576 573 A (RUEHENBECK WOLFGANG) 18 March 1986 (1986-03-18) figures 1-2B	1-3,9-12
X	US 2 906 696 A (P. W. GARBO ET AL.) 29 September 1959 (1959-09-29) figure 2	1-3,9-12

☐ Further documents are listed in the continuation of box C.   ☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
2 August 2001	09/08/2001

Name and mailing address of the ISA	Authorized officer
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Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
WO 0069552	A	23-11-2000	NL	1012082 C	21-11-2000
			AU	4955400 A	05-12-2000
US 5114700	A	19-05-1992	AU	581103 B	09-02-1989
			AU	6299886 A	26-03-1987
			CA	1277822 A	18-12-1990
			ZA	8607101 A	27-05-1987
US 4576573	A	18-03-1986	DE	3248502 A	05-07-1984
			AT	26930 T	15-05-1987
			DE	3371294 D	11-06-1987
			EP	0115019 A	08-08-1984
			JP	59173129 A	01-10-1984
US 2906696	A	29-09-1959	NONE		